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AN EVALUATION OF THE  
COOKING AND  
HOMOGENIZATION  
CHARACTERISTICS OF  
VARIOUS UNMODIFIED  
STARCHES

FRANK JOSEPH CLARKE

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AN EVALUATION OF THE COOKING AND HOMOGENIZATION CHARACTERISTICS  
OF VARIOUS UNMODIFIED STARCHES

A THESIS

Presented to  
the Faculty of the Division of Graduate Studies  
Georgia Institute of Technology

In Partial Fulfillment  
of the Requirements for the Degree  
Master of Science in Textiles

by  
Frank Joseph Clarke  
August 1952



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CHARACTERISTICS OF VARIOUS UNMODIFIED STARCHES



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## SUMMARY

The cooking and homogenization characteristics of six unmodified starches have been determined. These starches included corn, rice, tapioca, sago, wheat, and rice. The study was confined to an evaluation of the viscosities of hot starch pastes prepared by cooking and by homogenization as well as the viscosity stabilities of these prepared pastes.

The Brabender Viscograph was used in the preparation of cooked pastes and the Logeman homogenizer was used in the homogenization studies.

Viscosities were measured with three types of viscometers, namely, a modified pipette type, a torsion type, and a sonic type.

The cooking cycle consisted of a 45 minute period during which the cold starch slurry was heated to  $197.6^{\circ}$  F. followed by a 75 minute period during which the temperature of the paste was held at  $197.6^{\circ}$  F. In the homogenization studies, the cold starch slurries were preheated to a temperature of  $188^{\circ}$  F. and then homogenized at homogenization pressures up to 4000 pounds per square inch.

In the case of cooked starch pastes, the linear relationship of log maximum viscosity to log starch concentration was found for the six starches studied. These results are in agreement with those found in earlier experiments. After two hours of cooking, the only starches which showed viscosity stability were corn and wheat.

In the case of homogenization, there is a rapid drop in the viscosity of the hot pastes as the pressure of homogenization is increased;



however, a leveling of viscosity occurs at pressures in excess of 1500 pounds per square inch. As the pressure of homogenization was increased, there was a marked increase in the temperature of the preheated slurry during homogenization. Increases of temperature ranged from  $1^{\circ}$  F. at low pressures to  $16^{\circ}$  F. at 4000 pounds per square inch.

The viscosities of starch pastes prepared by homogenization at pressures exceeding 1500 pounds per square inch were considerably lower than those of starch pastes prepared by cooking.





## CHAPTER I

### INTRODUCTION

Before weaving yarn into cloth it is often necessary that a protective coating be applied to the yarn so that the strains and abrasions of weaving will not damage the yarn. The application of this protective coating is called slashing and the mixture used is known as size. Various mixtures are used depending on the nature of the fibers from which the yarns are spun. In the case of cotton, a mixture of starch, with gums, waxes, and oils added to impart special characteristics to the protective film, is used. For this film to be successful it must coat each thread completely and evenly without leaving uncoated portions or thick globs of size which would lead to weaving difficulties.

Starch, which constitutes the major portion of a size mixture, is insoluble in cold water and, therefore, it is necessary to convert it into a homogeneous, fluid form which can be applied to the yarn. Two methods are used to accomplish this:

- (a) Chemical modification, for example, by chlorination
- (b) Mechanical modification, by heat, agitation, or homogenization

In this study, the effects of heating, agitation, and homogenization on starch paste viscosity and stability have been evaluated. The effects of heating and agitation were studied with the Brabender Viscometer to simulate kettle cooking.

In kettle cooking, the unmodified starch is mixed first with water and then heated until it reaches the gelatinization temperature of



the starch. At this point the granular structure of the starch is distended to its maximum by the entrance of water into the vesicles. This together with the mechanical agitation accomplished by constant stirring causes the rupturing of the vesicles after which the soluble portion of the starch is released into the mixture and a decrease in viscosity occurs. The granules, being of various sizes, swell at varying rates and the amount of rupturing at any one temperature represents only a portion of the granules actually present. Therefore, the mixture continues to be viscous beyond the maximum viscosity point and the viscosity steadily decreases until the majority of the vesicles have been ruptured and a level viscosity is reached. The time required for this cooking varies with the starch. For comparative purposes, a two-hour cooking cycle was used in these experiments since this is typical of the cycle required for the stability of pearl corn starch pastes. Pearl corn represents the major starch used by the cotton textile industry for warp size.

Once the stable viscosity has been reached, the mixture, now called size, is ready for use. The quantities needed for slashing are drawn off and the remainder is stored at a constant temperature with mild agitation until needed. Since the size shows decreased protective efficiency after prolonged storage, only enough size for immediate use is usually prepared and new batches are added as required.

The method described above is called kettle cooking because a large size kettle containing heating coils or steam jets is used. It is the oldest of those methods now in use but it has the drawback of a long period of preparation with its attendant costs in labor and power. In addition, there is some question as to the actual stability of the size



prepared because of the excessive heating effects of live steam being injected into the slurry as it is cooked, together with the fact that not all the vesicles may have been ruptured causing a poor size to be made.

A newer method attempts to overcome these drawbacks by substituting mechanical shearing action for heating to rupture the starch vesicles thereby making it possible to reach a stable viscosity quickly while insuring complete destruction of the granular structure of the starch. This method is called homogenization. In practice the starch is mixed with cold water and heated to a temperature above its gelatinizing temperature. The heated slurry is then forced through an orifice which ruptures the vesicles thereby converting it into size. The heat generated by the homogenization pressure is sufficient to raise the temperature of the size to that desired for use. From the homogenizer the size passes to storage kettles where it is stored at a constant temperature with stirring in the same manner as is kettle cooked size. Homogenization under ideal conditions destroy the vesicular structure of the starch and there are no intact vesicles to rupture during storage as may occur with kettle cooked size.

The conversion of the hot slurry into finished size by homogenization is virtually instantaneous, being limited only by the capacity of the pump and pipe used to pass the mixture through the homogenizer. Therefore, it is possible to have a continuous operation and prepare only a quantity sufficient for immediate use without the long cooking cycle of the kettle cooking method. This results in lower labor and power costs while yet insuring that a stable size will be produced.



In this study, size prepared from starch and water only was used and no attempt was made to evaluate various other additives used in commercial size preparations.





## CHAPTER II

### EXPERIMENTAL PROCEDURE

Six unmodified starches were used in this study. A single one hundred pound bag of each starch was obtained and all experiments concerning that starch were made with portions drawn from that bag. The starches used were corn, tapioca, sago, rice, wheat, and potato. These six starches represent those most commonly used in the textile industry. All six bags were stored together under the same conditions of temperature and relative humidity.

To insure a thorough evaluation of the starches, three types of viscometers were used. The first, flow type, depends upon the measurement in seconds of the time of flow of a known quantity of liquid through an orifice.

The Ultra-Viscoson, a sonic type viscometer, was the second type used. In this instrument a probe, when inserted into a liquid, emits a very high frequency signal causing the liquid in contact with it to vibrate at a very high rate. These vibrations represent the rate of shear occurring at that time in the vicinity of the probe. The signal is passed from the probe through a cable to the instrument where the reading appears on a meter graduated in centipoises X grams/cubic centimeter. This makes it possible to read absolute viscosities directly from the meter, but since starch pastes are non-Newtonian in their characteristics, the readings are merely relative. The instrument provides a base range of 0 to 50 centipoises which was used in these ex-



periments. Additional ranges of ten, one hundred, and one thousand times the base range are possible if the viscosity being measured exceeds the base range.

The Brabender Viscograph was the third instrument used. It proved to be the most versatile, for in addition to 'simulating'\* the kettle cooking cycle it also provided a written record of viscosity as a function of time as well as temperature. This record is made by a recording kymograph which is connected through a spring to the resistance measuring rod extending down into the bowl containing the starch suspension. The bowl rotating at a constant speed of 75 RPM pushes liquid against the pins of the measuring rod extending down into it. This pressure against the pins causes the resistance measuring rod to rotate against the pressure of its spring. The amount of this rotation is recorded on the chart in arbitrary units, called Brabender Units. Therefore, the higher the apparent viscosity being measured, the greater is the rotation of the pins and, hence, the higher the Brabender Unit reading.

The spring furnished with the Viscograph was calibrated for the viscosity range of size, and the concentration of starch on a dry basis used in these experiments were chosen to cover the entire range of the Viscograph.

The "kettle cooking" cycle was simulated in the Viscograph by mixing about 200 milliliters of water with the quantity of starch to be

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\* In the normal kettle cooking procedure, live steam is employed whereas, in the Brabender unit, heating was accomplished by radiation only.





Figure 1. Brabender Viscograph with Bowl, Resistance Measuring Rod, and Timer Removed to Show Details of Construction.



tested. The volume was measured in a graduated cylinder and sufficient water then added to bring the total volume to 450 milliliters. The suspension formed was stirred for one minute with an electric stirrer. The bowl, resistance measuring rod, and coverplate from the Viscograph were removed and weighed. The suspension was then poured into the bowl and the gross weight obtained. The bowl and its contents were replaced in the Viscograph and the thermoregulator set to rise from 25 to 92° C.

Before starting the instrument to cooling coil was secured in the down position and the timer was set for 45 minutes to prevent overheating of the liquid. Upon reaching 92° C. the temperature was held constant for the remainder of the two hour cycle. Then the bowl and its contents were immediately removed and reweighed to obtain the moisture loss during the cycle.

The series of experiments of "kettle cooking" were divided into two groups:

(a) Those in which the slurry was heated from 25° C. to 92° C. after which it was kept at that temperature for an additional 75 minutes making a total cooking cycle of 120 minutes. During the entire cycle slow agitation at 75 RPM was maintained.

(b) Those in which the cycle described above was used but, in addition, cold water was allowed to flow through the cooling coil alternately with heating. This resulted in a series of heating and cooling steps as regulated by the temperature of the slurry

Group (a) series was performed on all six starches.

Group (b) series was performed on corn and tapioca only.

To reproduce the homogenization cycle used commercially, a fifty





gallon per hour capacity Logeman homogenizer was used. The cold slurry was prepared in a standard 100 gallon Lowell size kettle by mixing the quantity of starch to be used with about  $2/3$  of the total volume of water to be used. Then the resulting volume was measured and sufficient water to bring the volume to one pound of starch per gallon of finished slurry was added. Agitation by stirring was started as the slurry was prepared and continued while the slurry remained in the kettle.

From the kettle the slurry was pumped through a heat exchanger similar to the Girdler Corporation's Votator. This exchanger was built by the Georgia State Engineering Experiment Station for an earlier series of experiments and it proved well suited to the work.

In the heat exchanger the cold slurry was brought to  $87^{\circ}\text{C}$ . ( $188^{\circ}\text{F}$ .).

The steam to heat the heat exchanger was regulated by a Powers Accritem compressed air operated valve. This valve was actuated by an Invar thermobulb located in the flow line just beyond the heat exchanger so that any changes in temperature were readily compensated for by the regulator.

From the heat exchanger the hot slurry passed to the homogenizer and after homogenization was disposed of through a waste line. This line also contained a takeoff at which samples for the Brabender and the flow-type viscometric tests were collected.

The initial test in each homogenization series was made without pressure on the slurry as it passed through the homogenizer. As the size left the homogenizer its viscosity was measured by the Ultra-Viscoson and the flow-type viscometer. The initial and final tempera-



tures were recorded.

The homogenization experiments were made while holding the initial temperature, i.e., the temperature to which the slurry had been heated in the heat exchanger, constant at  $37^{\circ}$  C. ( $188^{\circ}$  F.) and varying the pressure of homogenization.

After four minutes of operation without pressure, 250 pounds per square inch of pressure was applied to the slurry and the same data recorded. Each four minutes thereafter the pressure was changed and readings made of its effect on viscosity and temperature rise. In addition, twice during each series, 450 milliliters of size were drawn off at the takeoff and a record of the viscosity and stability was made using the Viscograph.

The effect of cooling on the viscosity of homogenized potato, corn, and wheat starches was determined by taking Ultra-Viscoson readings on 450 Milliliter samples directly after homogenization and then allowing them to cool. The samples were stirred periodically and additional UV readings made during cooling.



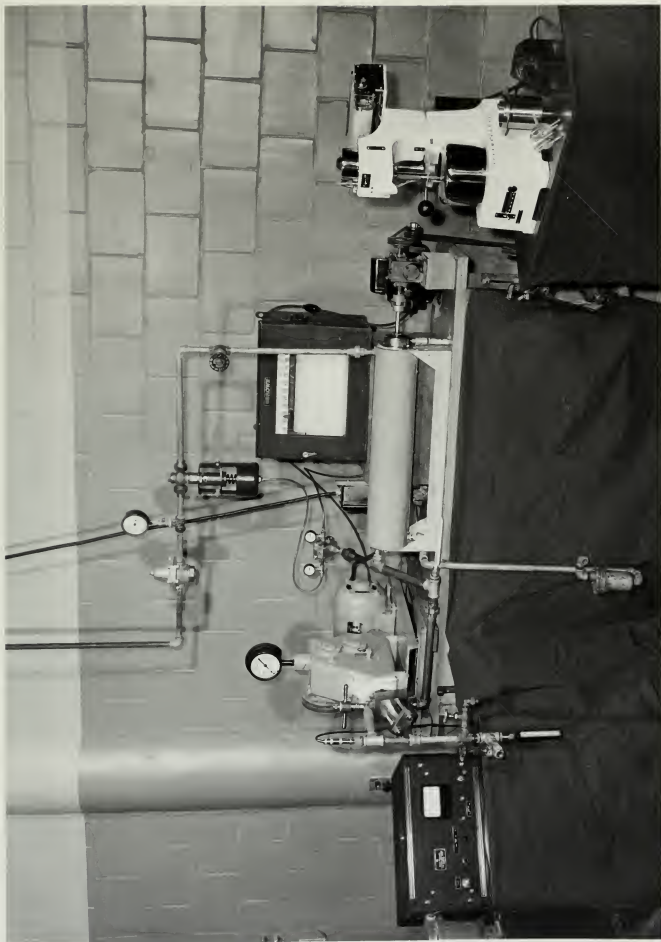


Figure 2. Experimental Apparatus Showing Viscograph, Heat Exchanger, Steam Regulator, Homogenizer, and Ultra-Viscoson with Its Probe in the Flow Line.





Figure 3. Rich-Roth Laboratories Ultra-Viscoson with Probe.





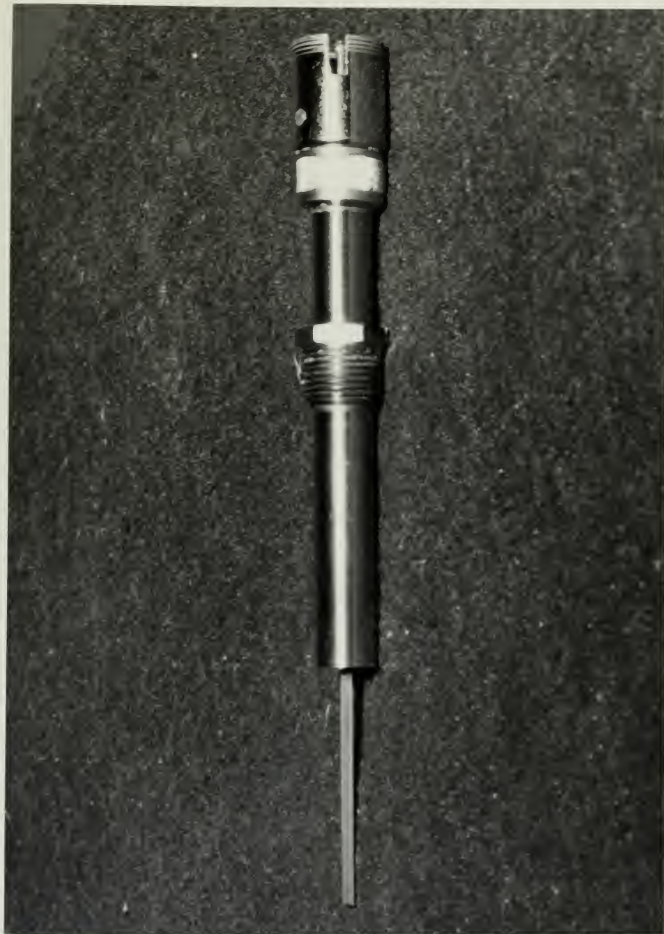


Figure 4. Close-up View of Ultra-Viscoseon Probe.



## CHAPTER III

## RESULTS AND DISCUSSION OF RESULTS

Figure 5 through 12 show that the straight line relationship between log starch concentration and log maximum viscosity in Brabender Units, discussed by Rask and Alsberg (2) and Anker and Geddes (3), holds true for all six starches in the concentrations used. In addition, this relationship holds for viscosities reached after two hours of preparation.

Table 1 gives the stability ratios computed by Higginbotham's method (4). These agree with those obtained with other types of viscometers.

In addition, it was found that small variations in concentration caused wide variations in Brabender readings. This agrees with the findings of Brown and Harrel (5).

It is to be noted that the starch concentrations are in all cases expressed on a dry weight basis. The moisture content of the starches was found to be:

Wheat	10.2%
Tapioca	10.4%
Corn	10.6%
Rice	10.6%
Potato	13.8%
Sago	14.2%

The average moisture loss during the two hour cooking cycle was ten per cent. Obviously, this moisture loss would mean that the viscosity actually measured after two hours of cooking corresponds to the viscosity



of a more concentrated paste than the concentrations indicated in Table 1.

Figures 13 and 14 show that slightly lower maximum viscosities and considerably lower level viscosities were obtained by "kettle cooking" corn and tapioca starches while using alternate heating and cooling throughout the cycle.

No explanation for this can be offered. However, it does not appear to be due to malfunctioning of the Viscograph or to unusual thickening, gel formation, etc. of the paste. The writer has been informed that similar results were obtained during experiments in the flour industry (6).

Tables 3, 4, and 5 show the stability of homogenized size as measured by the Viscograph. Two samples, one at a low pressure and the other at a high pressure of homogenization, were taken from each series of tests. The samples were placed in the preheated Viscograph and brought to a uniform temperature of  $92^{\circ}\text{C}$ . ( $197.6^{\circ}\text{F}$ .). It took approximately fifteen minutes to reach a level viscosity. However, this time lag is negligible when it is considered that heat was lost during transfer of the sample to the Viscograph and it was then necessary to bring the temperature to  $92^{\circ}\text{C}$ . The viscosity values obtained here were considerably lower than would have been obtained if comparable concentrations of the starches had been kettled cooked in the Viscograph for the entire cycle.

In "kettle cooking" the viscosity measurements made by the Ultra-Viscoson do not show the marked changes in viscosity indicated by the Viscograph. For example, the viscosity of 5.36% kettle cooked potato



starch was within a spread of 1.4 centipoises while the equivalent Viscograph readings were spread over 550 Brabender Units.

Figure 16 gives the Ultra-Viscoson measurements for sago, corn, rice, and potato starches. It is noted that the readings for sago show some deviation from the curve; however, this deviation is within the experimental error and is attributed to chance variation rather than to significant differences in viscosity.

As shown in Figure 17 and Table 2 it was found that, upon cooling, the viscosity of potato starch rose rapidly while the viscosities of wheat and corn showed a much slower rise during the same period.

Table 7 shows a comparison of the viscosities obtained by homogenization with those obtained by simple "kettle cooking" in the Brabender Viscograph. It is noted that the homogenized starch partes have considerably lower viscosities than those prepared in the Brabender Viscograph.





## CHAPTER IV

## CONCLUSIONS

There is a definite relationship between the log viscosity in Brabender Units for cooked starches and the log of starch concentration. This relationship can be described by the equation:

$$y = k x^n$$

where y is the viscosity in Brabender Units; x is the starch concentration; n is the slope of the log-log plot.

This equation can be applied to both the maximum viscosities and to those reached after two hours of cooking.

Although corn and wheat starches when "kettle cooked" appear to reach a stable viscosity in two hours of cooking, a longer period would be required for tapioca, sago, rice, and potato.

In the case of homogenized size, the size is at a stable viscosity as it leaves the homogenizer. However, the viscosity of the homogenized size is considerably lower than that obtained by "kettle cooking".



## APPENDIX



Table 1. Viscosity and Stability of Kettle Cooked Starch As Measured by Viscograph

% Starch By Weight Starch (Dry Basis)	Viscosity (at 197.6° F.) in B.U.				Stability Ratio*	
	Max Point	After 30 Mins.	60	75	30/Max.	60/30
Corn	4.92	125	110	110	.88	1.00
	5.90	260	200	200	.77	1.00
	6.88	430	325	300	.76	.92
	7.93	575	455	360	.79	.96
Tapioca	8.92	855	470	450	.55	.97
	4.83	140	130	130	1.00	.93
	5.75	260	240	225	.92	.94
	6.70	470	450	440	.91	.99
Wheat	7.67	740	590	540	.80	.92
	8.12	850	595	560	.70	.94
	4.87	45	40	40	.89	1.00
	5.79	85	70	65	.82	.93
Potato	6.76	175	115	110	.66	.96
	7.72	365	180	165	.49	.92
	8.69	565	350	270	.62	.77
	9.57	830	400	335	.48	.88
Rice	3.83	255	130	115	.50	.96
	4.79	555	220	165	.40	.75
	5.36	700	180	165	.26	.94
	5.74	880	200	175	.23	.90
Sago	4.85	85	80	80	.94	1.00
	5.77	190	140	135	.74	1.00
	6.73	295	200	175	.68	.88
	7.69	520	250	235	.48	.96
	8.65	805	360	295	.47	.82
	4.65	205	105	70	.52	.67
	5.23	315	160	100	.51	.63
	5.53	385	180	105	.47	.58
	6.46	500	260	175	.52	.67
	7.38	920	400	260	.44	.65

\*Stability Ratio - A Comparison of the Amount of Change in Viscosity Between Two Selected Points in the Cycle.



Table 2. Effect of Homogenization Pressures on the Viscosity of Homogenized Corn Starch When Initial Temperature is Constant.

Time in Minutes	Homog. Press. in p.s.i.	U.V. Reading in Centipoises x g/cc.	Initial Temp. of.	Final Temp. of.	Flow-Type Viscometer in Secs.	Brabender Viscograph in B.U.
0	0	9.0	188	186	NF*	--
4	250	8.0	188	189	NF*	--
8	500	7.5	188	189.5	25.0	220
12	750	7.0	188	191	12.0	--
16	1000	7.0	188	194	9.5	--
20	1250	6.8	188	194	8.9	--
24	1500	6.75	188	197	8.4	--
28	2000	6.5	188	200	9.2	110
32	2500	6.0	188	206	6.3	--
36	3000	6.0	191	207	7.4	--

\* No Flow Through Viscometer at This Pressure.





Table 3. Effect of Homogenization Pressure on the Viscosity of Homogenized Rice Starch When Initial Temperature is Constant.

Time in Minutes	Homog. Press. in p.s.i.	U.V. Reading in Centipoises x g/cc.	Initial Temp. °F.	Final Temp. °F.	Flow-Type Viscometer in Secs.	Brabender Viscograph in B.U.
0	0	7.75	188	188	NF*	
4	250	7.0	188	190	NF*	145
8	500	6.5	188	192	NF*	
12	750	6.25	188	196	9.8	
16	1000	6.25	188	197	8.5	
20	1250	6.0	188	198	8.5	
24	1500	6.0	188	198	8.3	
28	2000	5.8	188	198	8.2	
32	2500	5.75	188	200	7.8	
36	3000	5.5	188	203	7.6	80
40	3500	5.5	188	207	7.8	
44	4000	5.25	191	210	7.8	

\* No Flow Through Viscometer at This Pressure.



Table 4. Effect of Homogenization Pressures on the Viscosity of Homogenized Sago Starch When Initial Temperature is Constant.

Time in Minutes	Homog. Press. in p.s.i.	U.V. Reading in Centipoises x g/cc.	Initial Temp. °f.	Final Temp. °f.	Flow-Type Viscometer in Sec.	Drabender Viscograph in B.U.
0	0	14.5	188	208	NF*	
4	250	12.5	188	207	NF*	
8	500	10.0	183	203	NF*	
12	750	9.5	189	208	NF*	
16	1000	9.25	189	208	NF*	255
20	1250	9.0	189	208	NF*	
24	1500	9.0	189	208	NF*	
28	2000	9.0	189	208	8.2	
32	2500	9.25	189	208	3.8	
36	3000	9.25	189	208	8.9	170
40	3500	9.25	189	208	8.5	
44	4000	9.0	191	209	8.4	



Table 5. Effect of Homogenization Pressure on the Viscosity of Homogenized Potato Starch When Initial Temperature is Constant.

Time in Minutes	Homog. Press. in p.s.i.	U.V. Reading in Centipoises x g/cc.	Initial Temp. of.	Final Temp. of.	Flow-Type Viscometer in Secs.	Brabender Viscograph in B.U.
0	0	15	188	188	--*	--*
4	250	--	--	--	--	--
8	500	14	188	192	--	--
12	750	--	--	--	--	--
16	1000	12.5	188	197	--	--
20	1250	10.2	189	199	--	--
24	1500	10.5	189	199	--	--
28	2000	8.5	189	200	--	--
32	2500	8.0	190	204	--	--

\* No Readings Made with Flow-Type Viscometer or Viscograph for This Starch.



Table 6. Effect of Cooling on Homogenized Starch Viscosity.

Temp. °F.	UV Reading Centipoises	Starch	Homo. Pressure p.s.i.
199	10.5	Potato	1500
182	13.2	"	1500
152	15.5	"	1500
130	29.5	"	1500
197	6.75	Corn	1500
176	8.4	"	1500
165	13.3	"	1500
130	17.0	"	1500
195	4.75	Wheat	1500
180	5.0	"	1500
160	8.0	"	1500
140	9.5	"	1500
126	13.0	"	1500





Table 7. A Comparison of the Viscosities of Homogenized and Kettle Cooked Starch Size.

Starch	% Starch (Dry Basis)	Homogenization Pressure (p.s.i.)	Viscosity Homogenized Starch (B.U.)	Viscosity Kettle Cooked Starch* (B.U.)	Difference (B.U.)
Corn	10.8	500	220	800	580
	10.8	2000	110	800	690
Sago	14.0	1000	255	940	685
	14.0	3000	170	940	770
Rice	10.8	250	145	470	325
	10.8	3000	80	470	390

\* These values were determined by extrapolation of the log viscosity versus log starch concentration plots as shown in Figures 7, 8, and 10.







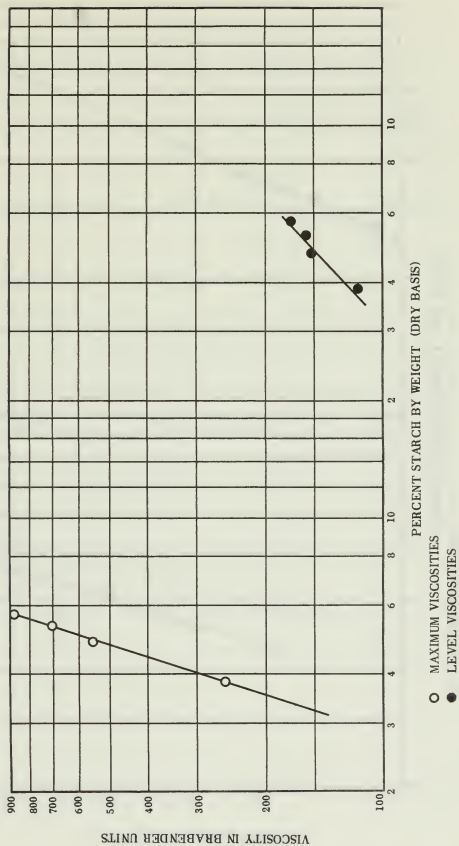


Figure 6. Maximum and Level Viscosities of Potato Starch as Measured by Viscograph.



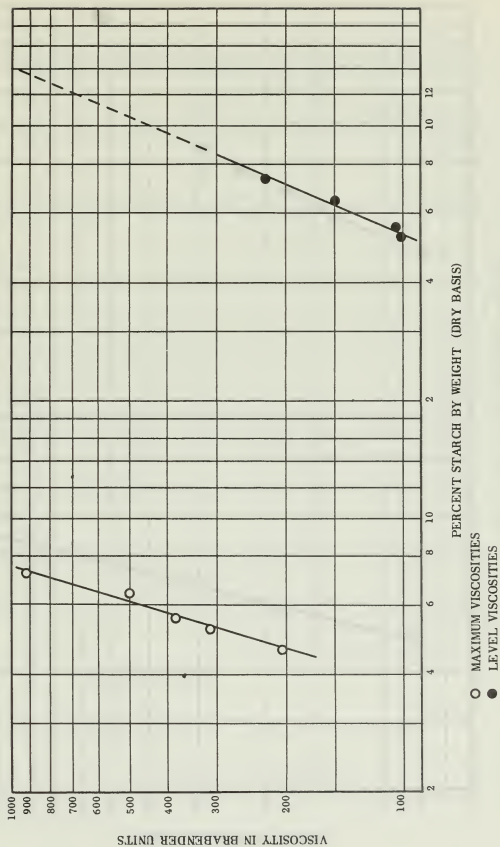


Figure 7. Maximum and Level Viscosities of Sago Starch as Measured by Viscograph.





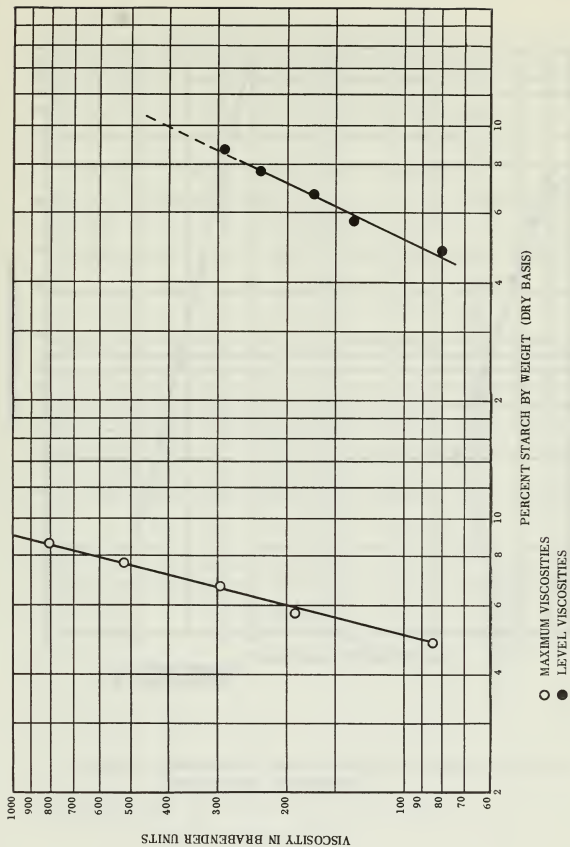


Figure 8. Maximum and Level Viscosities of Rice Starch as Measured by Viscograph.



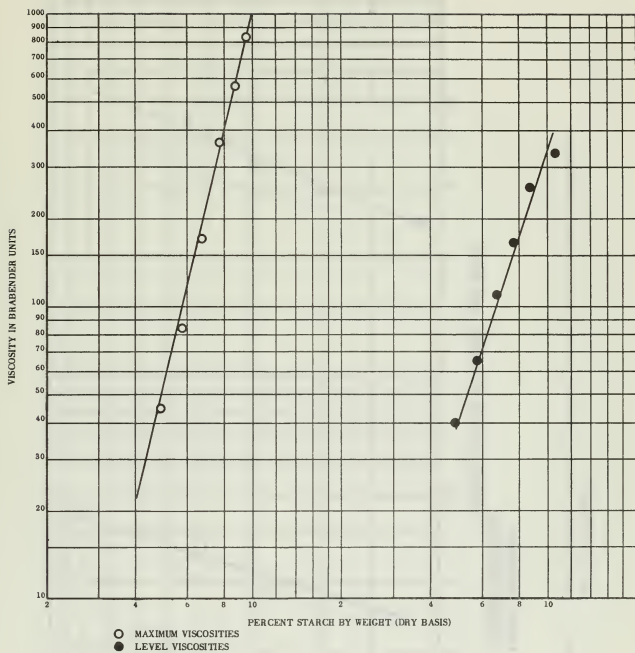


Figure 9. Maximum and Level Viscosities of Wheat Starch as Measured by Viscograph.



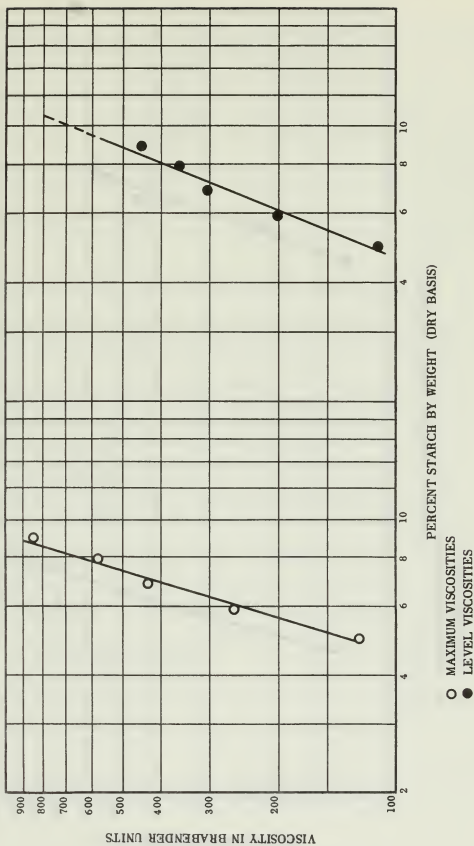


Figure 10. Maximum and Level Viscosities of Corn Starch as Measured by Viscograph.



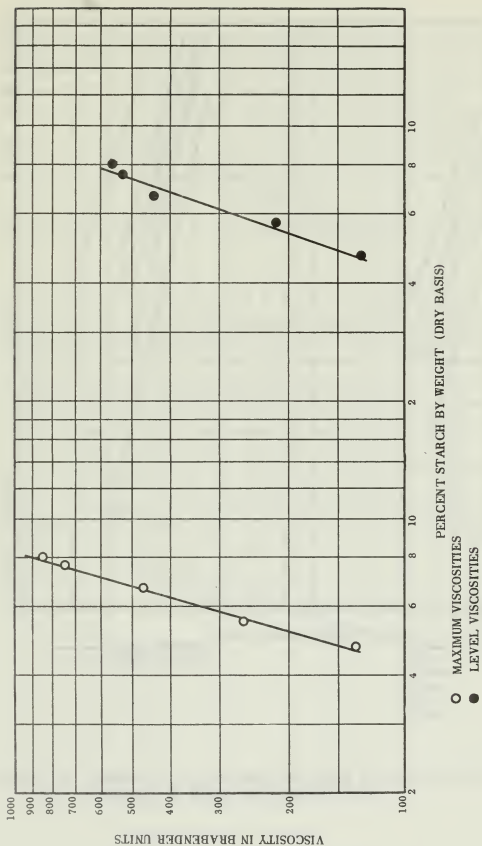


Figure 11. Maximum and Level Viscosities of Tapioca Starch as Measured by Viscograph.





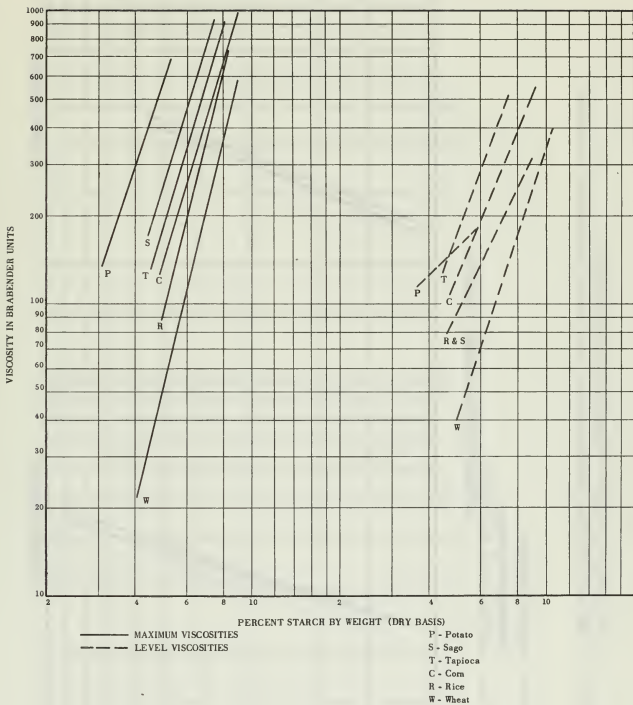


Figure 12. Summary of the Maximum and Level Viscosities of Six Unmodified Starches as Measured by Viscograph.



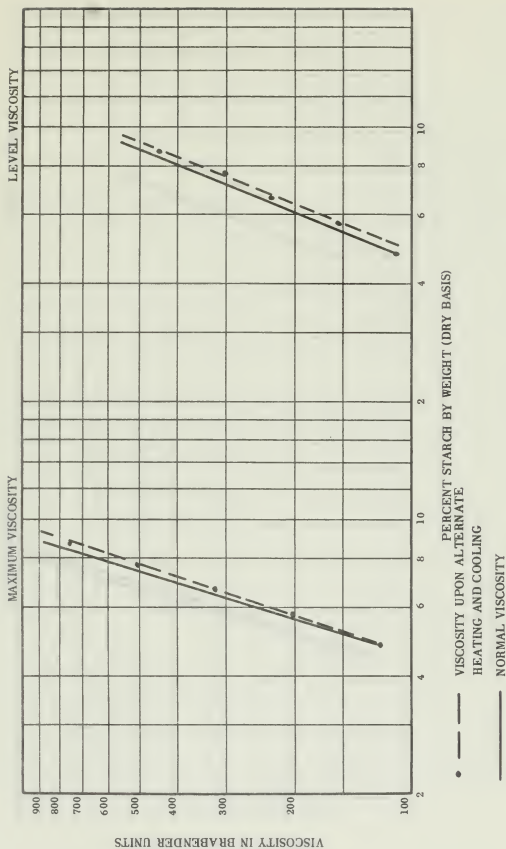


Figure 13. Maximum and Level Viscosities of Corn Starch Prepared by Alternate Heating and Cooling, as Measured by Viscograph.



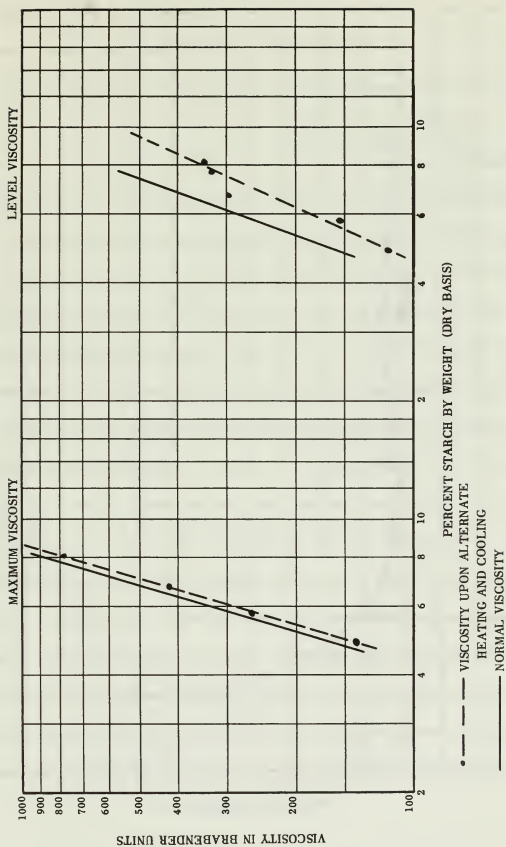


Figure 14. Maximum and Level Viscosities of Tapioca Starch Prepared by Alternate Heating and Cooling, as Measured by Viscograph.



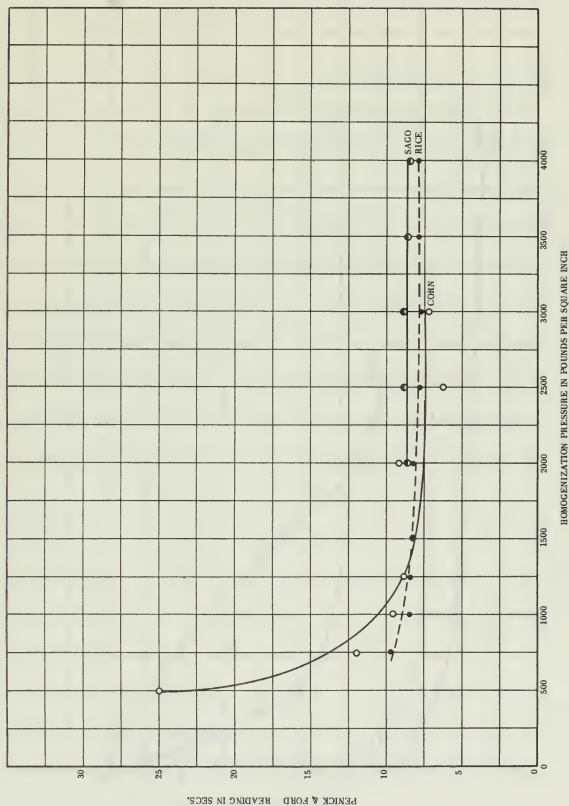


Figure 15. Effect of Homogenization Pressure on the Viscosity of Sago, Rice, and Corn Starches, When Initial Temperature is Constant, as Measured by Penick and Ford Viscometer.





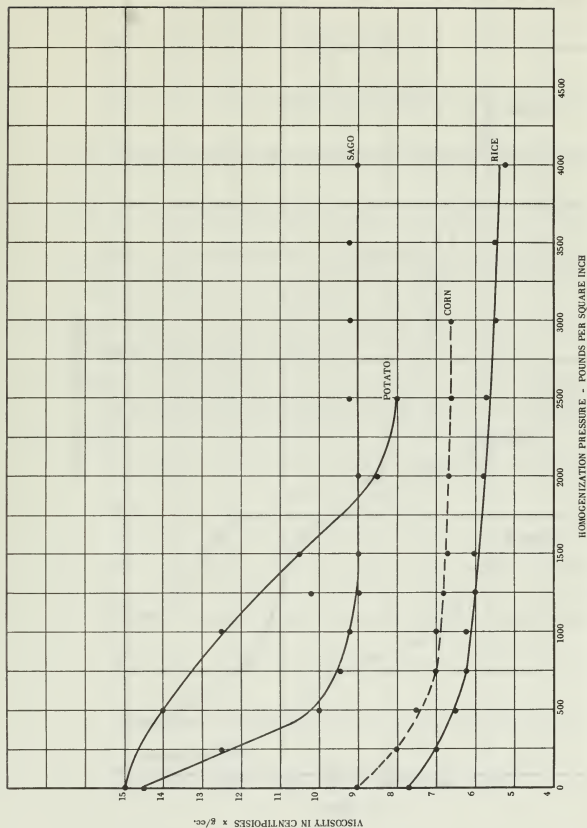


Figure 16. Effect of Homogenization Pressures on the Viscosity of Sago, Corn, Rice, and Potato Starches, When Initial Temperature is Constant, as Measured by Ultra-Viscosen Viscometer.



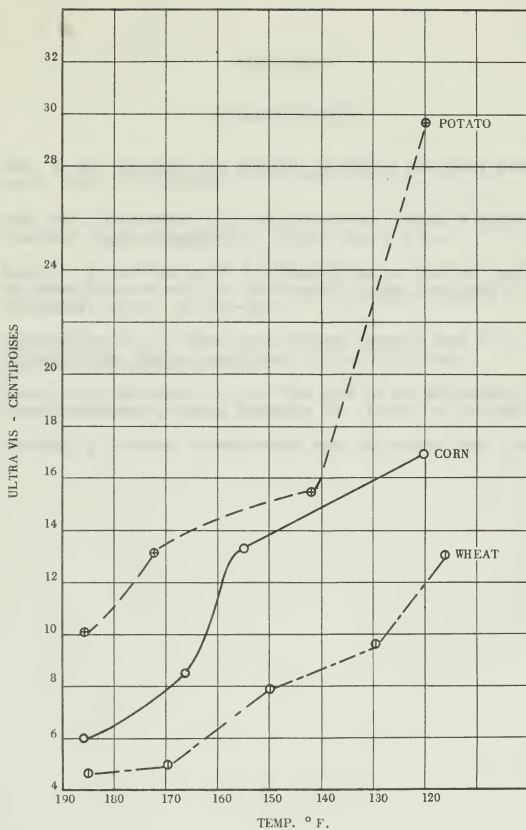


Figure 17. Effect of Cooling the Viscosity of Homogenized Wheat, Corn and Potato Starches.



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